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Optimization of Novacron Blue 4R (NB4R) removal by adsorption process on activated carbon using response surface methodology

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ABSTRACT

The objective of this study was to optimize the removal of Novacron Blue 4R (NB4R) dye from aqueous solution by adsorption on activated carbon using response surface methodology, through Box–Behnken design. The effect of process parameters such as pH, initial dye concentration and adsorbent concentration was studied and optimal conditions of NB4R removal were defined. The results showed that the adsorbent concentration was the most important parameter that affects the dye removal efficiency (CR%). The best condition for predicted maximum NB4R removal was found to be at pH 7, initial dye concentration of 62.5 mg/L and adsorbent concentration of 15 g/L. In these conditions, predicted and experimental CR% were 96.24% and 93.63%, respectively. Adsorption using activated carbon was proved effective for textile dye removal.

Keywords: Novacron dye; Adsorption; Activated carbon; Optimization; Response surface methodology; Box–Behnken

1. Introduction

It was considered that textile industries are among the most polluting industries in term of complexity of rejects and chemicals used in the dyeing process and in term of the volume of water consumed [1]. According to some studies, 700,000 tones and 10,000 different types of dyes and pigments are being produced annually across the world [2,3] and 15% of them are released into wastewaters [4]. In the aquatic ecosystem, the presence of dyes causes the depletion of the dissolved oxygen and a great harm on the food chain; by the inhibition of the photosynthesis process because they interfere with the transmission of sunlight [5]. In addition, they can be toxic, carcinogenic and mutagenic to the biocenosis [6,7].

Anthraquinone is considered as one of the major groups among reactive dyes. It has a carbonyl chromophore group (>C–O) on a quinone nucleus. The general formula derived from anthracene shows that the chromophore is a quinone ring on which hydroxyl or amino groups can be attached. The color of this group of dye is related to the anthraquinone nucleus and modified by the type, number and position of substituents [8]. Linked to the high solubility of reactive dyes, a large amount (10%–50%) is released into the environment [9,10]. The presence of aromatic rings in chemical structure of reactive dyes favors a high stability against light, oxidants and biological degradation [11]. The accumulation of anthraquinones in the environment leads to the destruction of aquatic life and causes serious health risk to human-like respiratory diseases and allergic dermatoses [12,13], change in immunoglobulin levels [14], the increased risk of colon and rectum cancers [15], high risk of contracting bladder cancer [16], genotoxicity [17] and teratogenic potential [18].

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It is of great importance to provide and develop wastewater treatment for minimizing the impact of these effluents. There are various methods available for wastewater treatment such as membrane filtration [19], coagulation–flocculation [20], advanced oxidation [21], photodegradation [22], electrocoagulation [23] and biological treatment [24].

Although, these methods have been widely applied, they have some disadvantages. Biological processes are less efficient in degrading synthetic dyes as reactive dye [25,26] and the breakdown of reactive dyes by anaerobic degradation leads to the formation of aromatic amines, which may be more toxic than the dye molecules as such [27]. Electrocoagulation and chemical coagulation cause extra pollution due to the undesired reactions in treated water and produce large amounts of sludge [28] membrane filtration, advanced oxidation and photodegradation are very costly [29–31]. All of these crucial limitations lead to the necessity of an effective and easily approach for textile effluents treatment.

Adsorption is a particularly effective method for wastewater treatment [32,33]. During this phenomenon, the pollutant is transferred from the liquid phase to the solid phase. Adsorption mainly depends on the nature of the adsorbent, its porosity, the surface state, the extent of its total surface (internal and external) and the physicochemical conditions of the medium [34–36].

Different types of materials have been used like adsorbent to trait toxic molecules include dyes [37–40]. Activated carbon is the most commonly used adsorbent for color reduction [41]. It contains microcrystalline graphitic carbon plates. The pores are from a few angstroms to a few hundred angstroms in which the circulation is by diffusion.

Response surface methodology (RSM) is a series of mathematical and statistical techniques that can be used for investigating the effects of several factors at different levels, studying their interaction and then, developing, improving and optimizing processes [42]. It has, as an advantage, a reduced number of experiments.

Box–Behnken design is an independent, rotatable quadratic design. It consists of a central point and the middle points of the edges of the cube [43]. It enables calculations of the response function at intermediate levels and allows estimation of the system performance at any experimental point within the range studied. It was applied as experimental design strategies by different authors [44,45].

The focus in this work was to study the adsorption efficiency of the reactive dye Novacron Blue 4R (NB4R) by investigating the effect of each factor and finding optimal conditions using RSM.

2. Materials and methods

2.1. Chemical structure of dye

Reactive dye used for this study is NB4R (Fig. 1) (IUPAC name: tetrasodium 1,2-bis(4 fluoro-6-[5-(1-amino-2-sulfonatoanthraquinone-4-ylamino)-2,4,6-trimethyl-3-sulfonatophenylamino]-1,3,5-triazin-2-ylamino)ethane; purity 70%–80%; Mw: 1,401.202 g/mol) supplied by the Textile Industrial Company (SITEX), 5070 Ksar Hellal, Tunisia, from Merck (Germany).

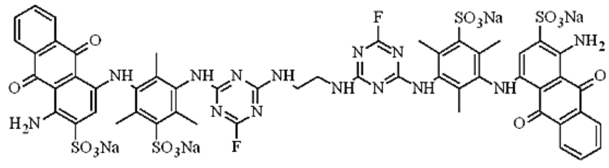

Fig. 1. Chemical structure of Novacron Blue 4R (NB4R).

2.2. Adsorbent

As an adsorbent, we used an activated carbon supplied from Textile Industrial Company (SITEX), supplied from "Chifeng City Fuyue Activated Carbon Factory, China". It is characterized by a density of 0.835, a pore volume of 0.34 cm^3/g, a pore diameter of 14 A and a Brunauer–Emmett–Teller surface area of 885 m^2/g.

2.3. Experimental condition

Dye solutions were prepared by dissolving proper mass of NB4R (62.5–127.33 mg/L) in distilled water. Experiments were performed using different levels of factors. For each experiment, 200 mL of NB4R solution was mixed with the adsorbent in a beaker and agitated for 24 h at ambient temperature with a NaCl concentration of 3.5 mg/L and an agitation speed of 50 rpm. The pH was adjusted by diluted sulfuric acid and sodium hydroxide using pH meter (370 pH meter Jenway). After 24 h, samples are removed and filtered through 0.45 μm filter (Whatman® Glass microfiber filters, Grade GF/C, from Sigma-Aldrich). All experiments were replicated with an experimental error of 3%. All chemicals used were supplied from Sigma-Aldrich, France.

2.3.1. Absorbance characteristics

Absorbance was measured using HACH Lange DR 3900 spectrophotometer, under a wavelength of 595 nm. The color removal efficiency (CR%) was calculated as follow [46]:

$$\text{CR\%} = \frac{\text{Abs}_{595}(t_0) - \text{Abs}_{595}(t_{24})}{\text{Abs}_{595}(t_0)} \quad (1)$$

where $\text{Abs}_{595}(t_0)$: measuring the absorbance at 595 nm at initial instant $t_0 = 0$ h; $\text{Abs}_{595}(t_{24})$: measuring the absorbance at 595 nm after 24 h $t_{24} = 24$ h.

2.4. Experimental design

Experiments were performed, through Box–Behnken design using three factors with different levels: initial dye concentration (62.5, 95 and 127.33 mg/L correspondent to an absorbance of 2, 3, and 4 a.u. at 595 nm), a pH (7, 9 and 11) and an adsorbent concentration (5, 10 and 15 g/L).

2.5. Statistical analysis

The experiments were statically designed using RSM which allows developing a statistical model showing the combined effect of the different independent parameters investigated on the response.

The results of NB4R removal using activated carbon were analyzed using the software MINITAB v 14.0.

The behavior of the adsorption process is clarified by an empirical second-order polynomial model equation. The reliability of the fitted model was justified through R^2 and analysis of variance (ANOVA) which is the amount of variance explained by the model. F value and P value (<0.05) were used to check the significance of the studied NB4R removal efficiency.

Residual plot, contour plot, main effect plot and interaction plot were also studied to determine the effect of each factor and the interactions between them.

3. Results and discussion

3.1. Response regression equation

In the present study, the chosen factors were initial dye concentration (mg/L), pH and adsorbent concentration (g/L). On the other hand, the experimental result of the response is CR%. The regression model equation is expressed as:

$$\begin{aligned} CR\% = &-13.8356 - 0.255\ (pH) + 13.903\ (Ac) + 8.1075\ (Abs) \\ &+ 0.9631\ (pH^2) - 0.2099\ (Ac^2) - 1.8575\ (Abs^2) \\ &- 0.9215\ (pH \times Ac) - 1.4875\ (pH \times Abs) \\ &+ 0.405\ (Ac \times Abs) \end{aligned} \quad (2)$$

$R^2 = 93.6\%$

where CR% is the color removal efficiency (%); Ac is the adsorbent concentration (g/L); Abs is the absorbance (a.u.) at 595 nm and R^2 is the regression coefficient.

The regression coefficient R^2 = 93.6% confirms the accuracy of the model (>90). We can assume that the model may be predictable and guaranties a good correlation between the parameters and the response. Additionally, we can deduce from coefficients of the equation that the adsorbent concentration (13.903) and the initial dye concentration (8.1075) are the most affecting factors on the response value of color removal (CR%).

3.2. Analysis of variance

The ANOVA is used for the determination of significant variables. Table 1 shows the obtained results of the statistical analysis ANOVA for the selected dye removal.

In Table 1, it is apparent that the sum of squares (SS) related to residual error value is negligible compared with the total sum of squares (244.27 << 3,791.79).

In our case, we noted that the P value for regression model equation is less than 0.05; it implies that the second-order polynomial model fits well to the experimental results. The regression adjusted average squares and the linear regression adjusted average squares were 8.07 and 2.89, respectively, for the response color removal (CR%). These parameters prove that the model accuracies are adequate to predict the performance of adsorption process.

3.3. Residual plot

The four-in-one residual plot displays of CR% are given in Fig. 2. This layout is useful for comparing the plots to determine whether the model meets the assumptions of the analysis. In addition to the mentioned criteria for evaluating the adequacy of the developed models, the difference between experimental and predicted responses (residuals) could be utilized for investigating the adequacy of the model graphically. Residuals are considered as unexplained variations by model and they will occur based on a normal distribution if the model is a good predictor [47].

Normal probability plots of residuals (Fig. 2(a)) showed that the points form a straight line so residuals are normally distributed. Residuals vs. fits plot (Fig. 2(b)) represent a random pattern of residuals on both sides of zero (0). According to these plots, residuals of CR% model have been randomly distributed. Histogram of residual (Fig. 2(c)) showed a symmetric distribution and residual vs. the order of the data (Fig. 2(d)) showed that the residual appear to be randomly scattered about zero. No evidence exists that the regression terms are correlated with one another. As a result, Fig. 2 shows that the model is adequate to describe NB4R removal CR% by RSM.

3.4. Contour plot

Contour plots of predicted NB4R removal efficiency are shown in Fig. 3. According to Fig. 3(a), it is observed that at fixed pH and by varying the adsorbent concentration from 5 to 15 g/L, the color removal efficiency increases substantially (from 50% to 90%). On the other side, at fixed adsorbent concentration and varying pH from 7 to 11, the variation seems to be weak. We then conclude that the effect of pH is more noticeable when the adsorbent concentration is high, but at lower adsorbent concentration, the effect of pH was not so high. This independence of pH factor could be explained by the fact that the rise of dye removal efficiency is attributed to the increase of adsorption surface area and the availability of more adsorption sites on adsorbent [48]. On the contrary,

Table 1
Analysis of variance (ANOVA) of the model of the color removal (CR%)

	Source	Regression	Linear	Square	Interaction	Residual error	Total
CR%	DF	9	3	3	3	5	14
	SS	3,547.52	2,975.56	180.49	391.47	244.27	3,791.79
	MS	394.169	141.368	60.164	130.49	48.854	
	F value	8.07	2.89	1.23	2.67		
	P value	0.017	0.141	0.39	0.159		

DF, Degrees of freedom; MS, mean square

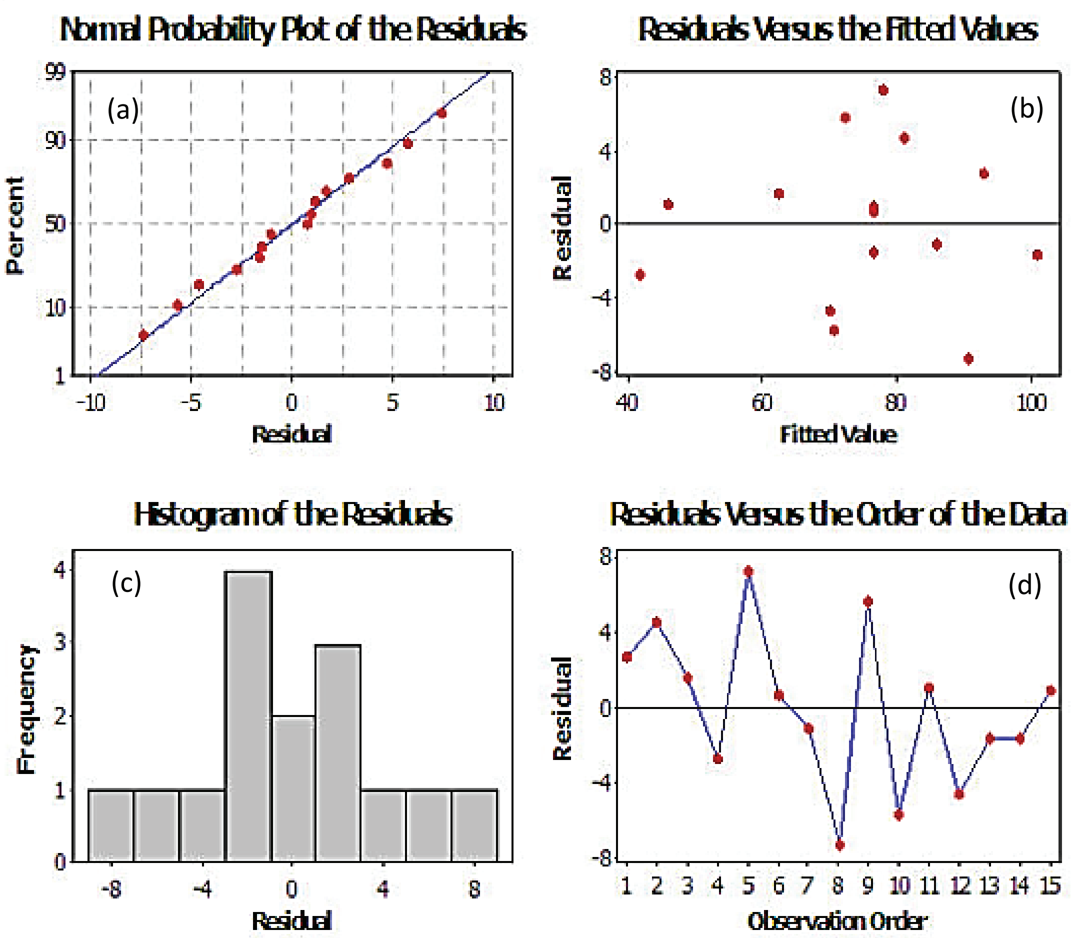

Fig. 2. Residual plots for response color removal CR%. (a) Normal probability plot of the residuals; (b) residual vs. the fitted values; (c) histogram of the residuals; (d) residual vs. the order of the data.

the effect of pH and the effect of adsorbent concentration are high at lower dye concentration, as it is shown in Figs. 3(b) and (c). That is due to the fact that at higher dye concentrations, there would be fewer available active sites on adsorbent than dye molecules in solution which lead to the decrease of dye removal efficiency (CR%) [49].

The study carried out by Radaei et al. [50] concerning the reactive blue 19 removal by residual-based activated carbon, proved similar observations.

Moreover, Sudamalla et al. [51] studying the adsorption of crystal violet by activated carbon prepared from mango kernel, reported that the color removal efficiency increased with decreasing the initial concentration of dye and increasing pH which is the case in Fig. 3(b).

Ghorbani and Kamari [52] studied the methyl orange adsorption by Fe-grafting sugar beet bagasse, proved similar results. They showed that adsorbent concentration is more effective than initial dye concentration and pH as appeared in Figs. 3(a) and (c).

3.5. Main effect plot

The mean changes that take place in the response (CR%) when the levels of each main factor were changed are presented in Fig. 4. In fact, when the line is horizontal, it means that no main effect is present. In the other word, when the line is not horizontal, it indicates the existence of a principal effect [53].

It has to be also noted that the statistical significance of a factor is directly related to the vertical line's length [54]. The larger the vertical line, the larger the change in the response when modifying the main factor from level –1 to level 1 [55].

Fig. 4 shows that the CR% increase with an average of 15% by varying the value of pH from 7 to 11; this could be explained by the fact that when pH increases, the hydroxyl ions concentration increases which leads to the rise of negative charges on the surface of adsorbent and since the dye is positively charged, the adsorption capacity increase consequently. It is also observed that CR% increases from 45% to 90%, when the adsorbent concentration varies from 5 to 15 g/L; that is due to the increase in availability of surface active sites on adsorbent [48].

In other word, it can be concluded that pH and adsorbent concentration have a positive effect on NB4R removal. However, initial dye concentration has a negative effect on dye removal. Moreover, from the length of the vertical lines, it can be observed that pH had the lowest effect on dye removal and the adsorbent concentration is the

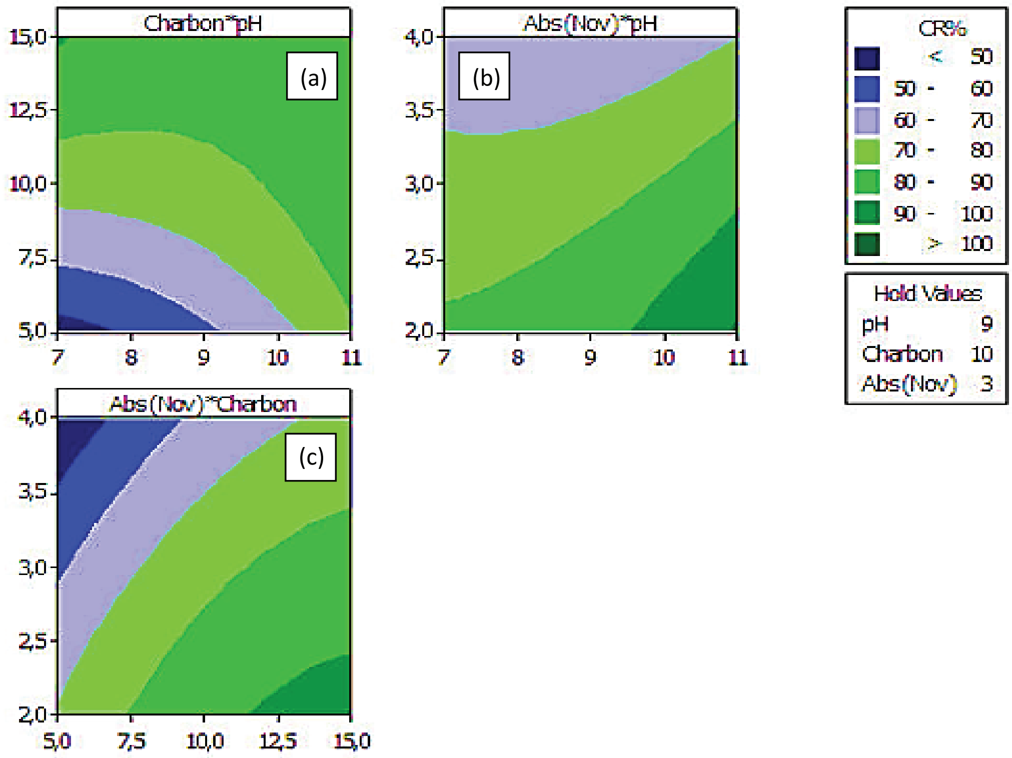

Fig. 3. Contour plots of Novacron Blue 4R (NB4R) removal. (a) Contour plots of activated carbon concentration (Y axis) with pH level (X axis); (b) contour plots of absorbance values of dye (Y axis) with pH level (X axis); (c) contour plots of absorbance values of dye (Y axis) with activated carbon concentration (X axis).

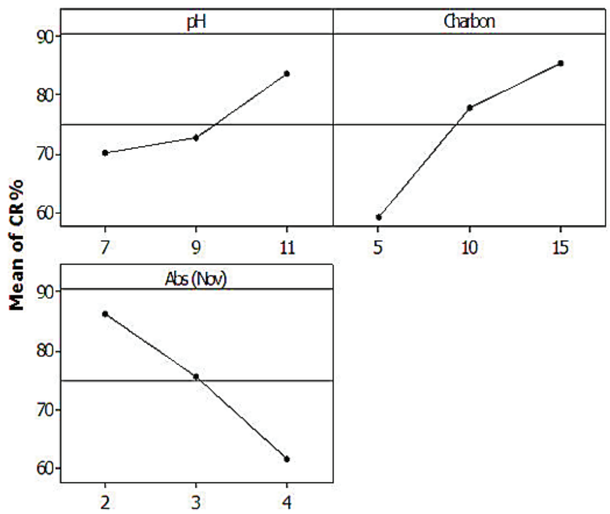

Fig. 4. Analysis of main effects plots of the response color removal (CR%).

most important parameter that significantly affects the dye removal efficiency.

Similar results were reported by Radaei et al. [50] who investigated in their study the adsorption of reactive blue 19 by pomegranate residual-based activated carbon. They reported that the important parameters affecting the color removal efficiency include pH, initial dye concentration and adsorbent dose. Their results showed that by increasing of initial pH and adsorbent dose, and decreasing of initial dye concentration, the dye removal efficiency improved.

3.6. Interaction plot

An interaction exists when the modification in the response from low to high levels of a parameter is dependent on the level of a second parameter. Graphically, an interaction is effective when the lines are not parallel [56].

The interaction effects of each factor on the dye adsorption are presented in Fig. 5. As can be seen, when the adsorbent concentration is increased from 5 to 15 g/L, color removal efficiency increased from 45% to 84% at pH = 7 and from 85% to 90% at pH = 11. We then conclude that there is an interaction between pH and the adsorbent concentration represented in the fact that the effect of the adsorbent concentration is high when pH is low but at higher pH, the effect of activated carbon is small. In addition, there is an interaction between the initial dye concentration and the adsorbent concentration. In fact, Fig. 5 shows that when initial dye concentration is increased from 62.5 to 127.33 mg/L, color removal efficiency decreased from 63% to 10% at an adsorbent concentration of 5 g/L and increased from 80% to 96% at an adsorbent concentration of 15 g/L. In other word, the effect of adsorbent concentration is higher at low initial dye concentration. Another interaction between pH and initial dye concentration is also shown in Fig. 5. It is observed that CR% decreased from 90% to 65% when initial dye concentration is increased from 62.5 to 127.33 mg/L at pH = 7 and increased from 68% to 98% at pH = 11.

Özbay et al. [57] have also found similar results in their study concerning the optimization of the reactive Remazol Yellow dye removal by carbon adsorption. They reported that color removal efficiency increase by increasing the adsorbent concentration and decreasing the initial dye concentration.

3.7. Optimized conditions

Fig. 6 suggests that the adsorbent concentration value should be kept as high as possible, as CR% increases monotonically with increasing the adsorbent concentration. On the contrary, the initial dye concentration should be kept low to optimize the response CR%. The optimal values of pH, initial dye concentration and adsorbent concentration are 7, 62.5 mg/L and 15 g/L, respectively. Color removal at these optimal conditions

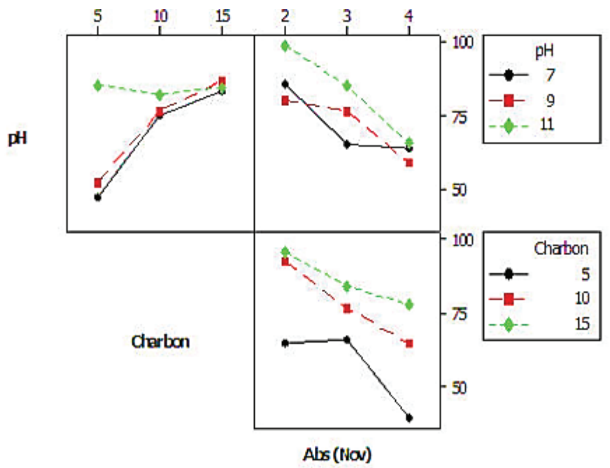

Fig. 5. Analysis of interaction plots of the color removal (CR%).

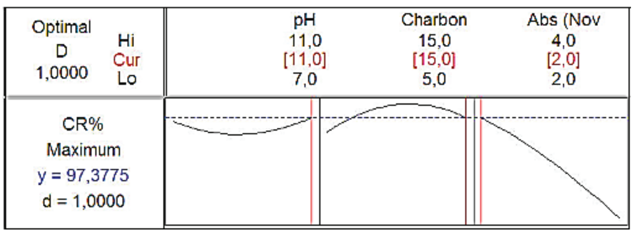

Fig. 6. Diagram of color removal CR% optimization.

Table 2
Comparison between experimental and predicted values using Box–Behnken design

	Predicted response	Experimental values
Global solution	pH = 7 Do(Nov) = 2 a.u.; [Nov] = 62.5 mg/L Carbon = 15 g/L	pH = 7 Do(Nov) = 2 a.u.; [Nov] = 62.5 mg/L Carbon = 15 g/L
Response CR%	CR% = 96.242%; desirability = 1	CR% = 93.63%

is CR% = 96.63% with a desirability *d* = 1. Experimentally, using the same conditions created by Minitab software, we obtained CR% = 93.63% (Table 2). Since the experimental and the predicted values are close, we could conclude that our model is globally validated but could be more improved.

Özbay et al. [57] investigating the optimization of the reactive Remazol Yellow dye removal by carbon adsorption found that the maximum color removal efficiency was obtained is 82.12% with 0.4 g/50 mL of adsorbent concentration at pH = 7.

Sudamalla et al. [51] showed that optimal conditions for the adsorption of Crystal Violet with activated carbon are reached at pH = 6, initial dye concentration of 50 mg/L and adsorbent concentration of 0.375 g/L with a decolorization efficiency of 89%. According to the study by De Luna et al. [58], using the Box–Behnken design, the optimal conditions obtained from the decolorization of Eriochrome black T were an initial dye concentration of 95 mg/L, an adsorbent concentration of 40 g/L and a pH 2 for a maximum CR% of 93.14%.

4. Conclusion

The RSM has been applied to create optimal conditions for NB4R removal by adsorption process. A factorial design and mathematical quadratic polynomial model of response surface were developed for NB4R removal. The regression model equation follows the experimental data with a good accuracy proved by R^2 = 93.6% which confirms that Box–Behnken design can be used for experimental design of dye adsorption by activated carbon. pH, initial dye concentration and adsorbent concentration are shown as an effective parameters on the color removal efficiency CR%. A maximum response of 96.242% was predicted for NB4R removal at low dye concentration of 62.5 mg/L, at high adsorbent concentration of 15 g/L and at pH 7. The good agreement found between observed and predicted values supports and confirms that the applied model is adequate to predict adsorption state.

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